

# Correlation between copper-activated pyrite flotation and surface species: Effect of pulp oxidation potential

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## Abstract

The effect of pulp oxidation potential, Eh, on the flotation of copper-activated pyrite at pH 9.0 in the presence of xanthate was investigated. It was found that pyrite recovery is at a maximum value when the Eh during conditioning is approximately 35 mV (SHE), but decreases at lower and higher Eh values. Solution and surface analytical techniques (EDTA extraction, and UV–visible and X-ray photoelectron spectroscopies) were used to explain the trends observed in the pyrite recovery with Eh. The increase in pyrite recovery with increasing Eh values has been attributed to an increase in xanthate adsorption at the pyrite surface while the decrease in recovery at high Eh values is the result of an increase in the proportion of surface iron oxidation species covering the hydrophobic species.

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## 1. Introduction

Iron sulphides, particularly pyrite, are often associated with copper sulphide minerals in ores. During grinding and conditioning, a variety of surface reactions can take place. For example, activation of pyrite can occur as a result of dissolution of copper species from chalcopyrite (Finkelstein, 1997; Chrysosoulis et al., 1992; Fuerstenau, 1982). These copper species form hydrophobic species on the pyrite surface with sulphide and collector, and therefore promote pyrite flotation, which subsequently lower the overall copper grade.

Pyrite in the presence of xanthate can only float in acidic to neutral pH conditions where dixanthogen, the oxidation product of xanthate, is stable (Fornasiero and Ralston, 1992). Pyrite flotation can also occur after

activation of pyrite with cupric species either added intentionally or dissolved from copper minerals present in the system. Several studies have investigated the activation of pyrite by copper (Weisener and Gerson, 2000a,b; Leppinen et al., 1995; Voigt et al., 1994; Wang et al., 1989; Girczys and Laskowski, 1985; Nicol, 1984; Bushell and Krauss, 1962). In alkaline pH conditions, Cu(II) adsorbs on pyrite as copper hydroxide, reacts with sulphide ( $S_2^{2-}$ ) at the pyrite surface and is subsequently reduced to a Cu(I)S species via sulphide oxidation to  $S_2^{1-}$  or polysulphide,  $S_n^{2-}$ . At high pH values and for a heavily oxidized pyrite surface, the rate of copper adsorption is low because copper has to diffuse through the surface iron hydroxide layer. Copper activation of pyrite does not involve an exchange of iron with copper, as it is the case for sphalerite where zinc is replaced by copper, and therefore is only limited to the top surface layer of pyrite. At a high copper concentration, copper hydroxide is also present on the pyrite surface. Xanthate reacts with surface copper sulphide and copper hydroxide, and forms Cu(I)–xanthate and

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dixanthogen thus rendering the pyrite surface hydrophobic (Laajalehto et al., 1999; Voigt et al., 1994). Pyrite flotation is reduced when its surface is oxidized, with iron sulphate and oxide/hydroxide being the major surface oxidation products in alkaline pH conditions (Fornasiero et al., 1992; Chander and Briceno, 1987; Buckley and Woods, 1987).

Pulp oxidation potential (Eh) controls the formation of these surface species responsible for pyrite flotation, polysulphide, elemental sulphur and xanthate but also sulphate and ferric oxide/hydroxide. For example, xanthate adsorbs on pyrite through an electrochemical reaction (Leppinen et al., 1995; Leja, 1982; Rao, 1971).

Infrared and X-ray photoelectron spectroscopies, and cyclic voltammetry have been used to study the effect of Eh on copper activation of pyrite and more particularly the species formed on the pyrite surface (Laajalehto et al., 1999; Laajalehto et al., 1995; Richardson et al., 1996).

In this study, we have used a range of solution and surface analytical techniques (EDTA extraction, and UV–visible and X-ray photoelectron spectroscopies) to identify and quantify the species formed on the pyrite surface at pH 9 as a function of Eh values with the aim to correlate these species with results of pyrite flotation.

## 2. Experimental section

### 2.1. Flotation experiment

All chemicals used were of analytical grade. The collector, sodium isopropyl xanthate (SIPX), was further purified by dissolution in acetone and recrystallization from petroleum ether using the methodology described by Montalti et al. (1991). The oxidation potential, Eh, in solution was measured with a platinum electrode. Pyrite was obtained from Huanzala Mine in Peru. Its chemical composition is reported in Table 1. Pyrite (100 g; 0.6–3.2 mm) was ground with 12 mild steel rods in a rubber lined-Galigher mill with 0.15 dm<sup>3</sup> of Adelaide water, 1000 g/t CuSO<sub>4</sub> and 750 g/t NaOH to produce a mill discharge particle size d<sub>90</sub> of 45 µm, a pH of 9.0 and an Eh of –250 mV (SHE). The pH was kept constant at 9.0 throughout conditioning and flotation with addition of NaOH. The conditioning times for the pyrite, SIPX and the frother (Dowfroth250) were 5.0, 2.0 and 1.0 min, respectively. Flotation experiments were performed with an Agitair flotation machine with a

total pulp volume of 1.5 dm<sup>3</sup> at an air flow rate of 3.0 dm<sup>3</sup>/min. The Eh was controlled during the conditioning step by purging nitrogen, air or oxygen to obtain the desired Eh value before collector addition.

### 2.2. Solution and surface analysis

Ethylene diaminetetraacetic acid (EDTA) was used to extract oxidation products (oxide, hydroxide and sulfate) from the pyrite surface (Rumball and Richmond, 1996; Kant et al., 1994). Two grams of the first flotation concentrate were mixed with 0.1 dm<sup>3</sup> of a 3% EDTA solution for 10 min and then separated by filtration. The EDTA solution was purged with high quality nitrogen gas before and during the extraction to prevent/reduce further mineral oxidation. The amounts of copper remaining in solution and surface metal oxidation products extracted by EDTA were measured by inductively coupled plasma mass spectroscopy by Amdel Pty. Ltd., Australia. The amount of copper adsorbed on pyrite was calculated from the amount of copper sulphate added and the amount of copper remaining in solution.

A CARY 1E spectrometer was used to measure the concentration of SIPX remaining in solution after contact with the pyrite sample. The molar absorptivity of xanthate is 17,500 cm<sup>–1</sup> dm<sup>3</sup> mol<sup>–1</sup> at a wavelength of 301 nm (Montalti et al., 1991).

A X-ray photoelectron spectrometer (XPS), Perkin Elmer Physical Electronics Division (PHI) 5100, with a MgKα X-ray source operated at 300 W was used to measure the species present on the pyrite surface just before flotation. A pass energy of 18 eV was used for all elemental spectral regions. The pressure in the analyser chamber was 10<sup>–7</sup> Pa. The energy scale was calibrated using the Fermi edge and the 4f<sub>7/2</sub> line (BE = 84.0 eV) for gold. Samples taken just before frother addition were washed with a pH 9.0 solution to remove any suspended colloid particles and were introduced immediately in the fore-vacuum of the XPS spectrometer as a slurry.

## 3. Results and discussion

### 3.1. Flotation experiments

Pyrite flotation in the absence of copper sulphate was very low (<10%) even in the presence of collector, and is mostly attributed to entrainment. A maximum flotation rate of pyrite at pH 9.0 was obtained at copper sulphate and SIPX concentrations of 1000 and 120 g/t, respectively. Higher copper sulphate concentrations produced a lower rate of pyrite flotation as a result of copper hydroxide precipitation on the pyrite surface as detected by XPS (Boulton et al., 2003). The flotation results of

Table 1  
Mineralogical composition of the pyrite sample

Elements	Fe	S	Cu	Pb	Si	Ca	Mg	Zn	Mn
%	45.6	52.0	0.12	0.05	0.255	0.26	0.025	0.07	<0.005

copper-activated pyrite as a function of pulp oxidation potential are shown in Fig. 1. Only the 0.5 min pyrite recoveries were reported as Eh increased quickly during flotation with addition of air or even nitrogen (gas flow rate was kept constant at 3.0 dm<sup>3</sup>/min during flotation). However, the same trend of flotation recovery with Eh was observed for longer flotation times. The recoveries at 0.5 min of flotation were found to be reproducible to  $\pm 2\%$ . Pyrite recoveries were very low (less than 10%) at negative Eh values ( $< 7$  mV) but improved sharply above 7 mV to reach 31% at 35 mV. However, with a further increase in Eh values, pyrite recovery decreased to a minimum of 8% at an Eh value of 265 mV.

Using a chalcopyrite electrode, Leppinen et al., 1998 observed that the flotation recovery of pyrite in three copper ores was maximum at Eh values of  $-50$  to  $0$  mV (SHE) and decreased at more reducing and oxidising conditions (Leppinen et al., 1998). According to these authors, the potentials measured with a chalcopyrite electrode are 70–90 mV lower than those measured with a platinum electrode. Therefore, their Eh values for maximum pyrite flotation correspond to 20–90 mV with a platinum electrode, which is in agreement with the value of 35 mV found in the present study with that type of electrode. Furthermore, if we compare the flotation edge for copper-activated pyrite of 7 mV (SHE) obtained in this study to the Eh values reported by Richardson and Walker (1985) for the flotation edge of various sulphide minerals, we can conclude that copper-activated pyrite in the present study behaves more like bornite in flotation (flotation edge at around 0 mV (SHE)).

Several studies have shown that the flotation of a sulphide mineral is related to the surface hydrophobicity of that mineral which in turn depends on the balance of hydrophobic (e.g., collector, polysulphide) and hydrophilic (e.g., metal oxide/hydroxide, sulphony) surface species (e.g., Boulton et al., 2003). The following results will attempt to correlate these trends in pyrite flotation

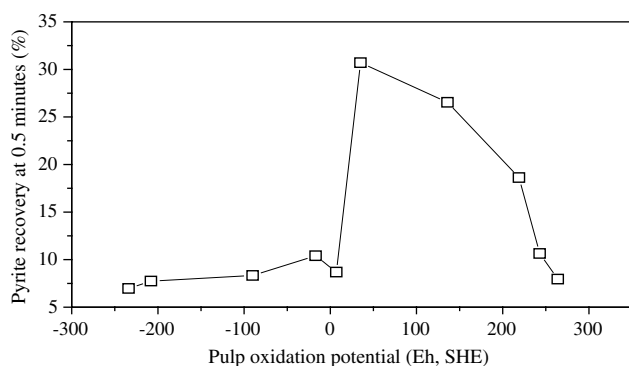


Fig. 1. Pyrite recovery after 0.5 min of flotation as a function of pulp oxidation potential (pH = 9.0; (copper sulphate) = 1000 g/t; (SIPX) = 120 g/t).

to the type and proportion of species present at the pyrite surface.

### 3.2. Solution and surface analyses

#### 3.2.1. Iron and copper species on the pyrite surface

Results in Table 2 show that the amount of Fe oxidation species extracted by EDTA at pH 9.0 from the pyrite surface increases with Eh value in agreement with earlier experimental results (Fornasiero et al., 1992) and thermodynamic calculation showing that ferric hydroxide is a stable species on the pyrite surface at positive Eh values (Garrels and Christ, 1965). This is also the case for the surface Cu oxidation species (detected by EDTA extraction); however their concentration is much less than that of the surface Fe oxidation species or the total amount of Cu adsorbed. This implies that most of the adsorbed copper is in a form that cannot be extracted by EDTA, probably as copper sulphide as previously observed in several studies (Weisener and Gerson, 2000a,b; Voigt et al., 1994). Furthermore, the total amount of copper adsorbed on pyrite appears to be independent of Eh in agreement with earlier results (Leppinen et al., 1995).

#### 3.2.2. Xanthate adsorption

Fig. 2 shows the UV–visible absorption spectra of SIPX in solution at pH 9.0 as a function of Eh and contact time with pyrite. The spectra are composed of two major peaks at 301 and 230 nm that are characteristic of xanthate while the minor peak at around 350 nm indicates that perxanthate, the decomposition product of xanthate, is formed under these conditions (Montalti et al., 1991). The spectra show that the absorbance of the 301 nm peak decreases with time indicating that with increasing Eh value more xanthate adsorbs on pyrite and at a faster adsorption rate. The amount of SIPX adsorbed on the pyrite surface at the start of flotation is shown in Fig. 3 as a function of Eh.

#### 3.2.3. XPS surface analysis

The proportions of elements (C, O, S, Fe and Cu) on the surface of pyrite samples collected just before frother

Table 2

Cu and Fe species present on the pyrite surface in the first flotation concentrate as a function of Eh (pH = 9.0; (copper sulphate) = 1000 g/t; (SIPX) = 120 g/t)

Eh, mV (SHE)	Cu species (kg/t of pyrite)		Fe species extracted by EDTA (kg/t of pyrite)
	Adsorbed	EDTA extracted	
–235			2.9
–60	0.36		
40		0.037	4.2
130	0.33		
265		0.064	7.2

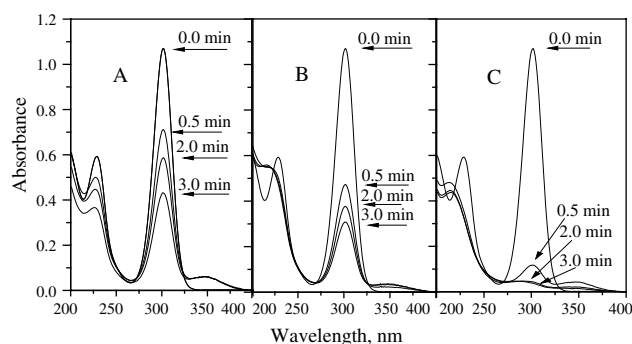


Fig. 2. Absorbance spectra of SIPX remaining in solution as a function of contact time with pyrite and at Eh values of, (A)  $-230$  mV, (B)  $35$  mV and (C)  $260$  mV (pH = 9.0; (SIPX) =  $6.09 \times 10^{-5}$  mol/dm<sup>3</sup>; (pyrite) =  $72.89$  g/dm<sup>3</sup>).

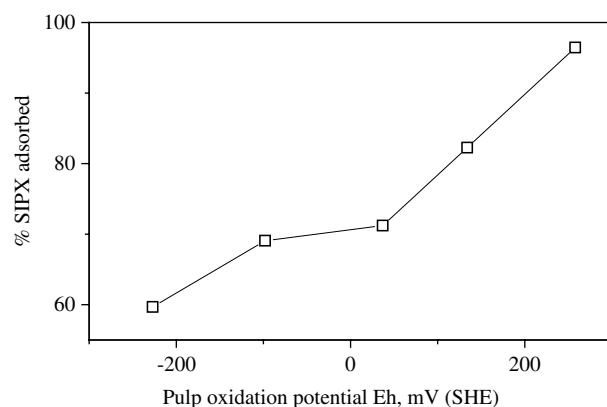


Fig. 3. Proportion of xanthate adsorbed at the start of flotation as a function of Eh (contact time with pyrite = 3 min; pH = 9.0; (SIPX) =  $6.09 \times 10^{-5}$  mol/dm<sup>3</sup>; (pyrite) =  $72.89$  g/dm<sup>3</sup>).

Table 3

Atomic proportions (%) measured by XPS of the elements present on the pyrite surface at negative and positive Eh values (pH = 9.0; (copper sulphate) =  $1000$  g/t; (SIPX) =  $120$  g/t)

Elements (%)	$-60$ mV (SHE)	$+130$ mV (SHE)
C (total)	<b>35.4</b>	<b>30.2</b>
C (xanthate)	5.6	6.7
C (hydrocarbon)	29.8	23.5
O (total)	<b>38.8</b>	<b>48.1</b>
S (total)	<b>9.7</b>	<b>6.5</b>
S (sulphide species)		
Surface $S_2^{2-}$	0.5	0.5
Bulk $S_2^{2-}$	5.8	3.5
Polysulphide $Sn^{2-}$	1.2	0.6
Elemental sulphur	1.7	1.0
S (S–O species)		
Fe (total)	<b>14.4</b>	<b>14.5</b>
Fe (sulphide species)	2.0	1.0
Fe (oxidised)	12.4	13.5
Cu (total) (as Cu(I))	<b>1.7</b>	<b>0.7</b>

addition at Eh values of  $-60$  and  $130$  mV (SHE) are shown in Table 3. The results indicate that more surface oxidation species (%O) are present at a positive Eh value than at a negative Eh value while the opposite is true for the proportion of carbon, sulphur and copper species (%C (total), %S (total) and %Cu).

More detailed information on the type of surface species can be extracted from the XPS spectrum by deconvoluting each spectrum into components. Several components are found in the C 1s XPS spectra shown in Fig. 4. The SIPX contributes to the C 1s spectrum with peaks at  $288.30$ ,  $286.46$  and  $284.60$  eV attributed to  $-C-(S)_2$ ,  $-C-(CH_3)_2$  and the two  $CH_3$  end groups, respectively with peak intensities 1:1:2 as the chemical structure of SIPX suggests. The other two peaks at  $284.60$  and  $286.09$  eV are due to adventitious hydrocarbon species, and C–C, C–H and C–O species, respectively (Fairthorne et al., 1997). No peak related to carbonate species is found at  $288.50$  eV (Wagner, 1994). The Fe 2p (2/3) spectra (not shown) are composed of only two peaks at  $711.2$  and  $706.6$  eV. The former is attributed to iron oxide/hydroxide while the latter

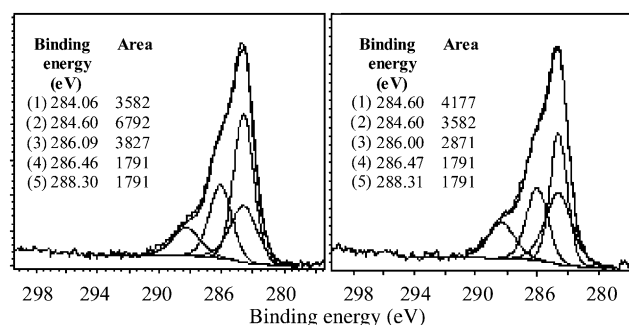


Fig. 4. C 1s XPS spectra of pyrite at Eh values of (left)  $-60$  mV and (right)  $130$  mV. The binding energy and area of all the peaks used in the fitting of the XPS spectra are shown in the insert (pH = 9.0; (CuSO<sub>4</sub>) =  $1000$  g/t; (SIPX) =  $120$  g/t).

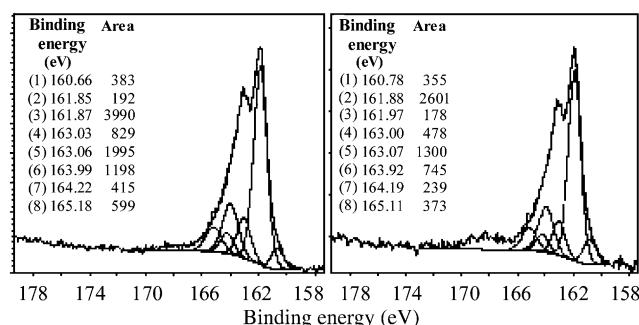


Fig. 5. S 2p XPS spectra of pyrite at Eh values of (left)  $-60$  mV and (right)  $130$  mV. The binding energy and area of all the peaks used in the fitting of the XPS spectra are shown in the insert (pH = 9.0; (CuSO<sub>4</sub>) =  $1000$  g/t; (SIPX) =  $120$  g/t).



belongs to iron in pyrite (Fairthorne et al., 1997). Each component in the S 2p spectra of Fig. 5 is composed of two peaks (doublet) separated by around 1.2 eV and with the intensity of the low binding energy peak double that of the high binding energy peak. The four doublets at 160.7, 161.9, 163.0 and 164.0 eV represent  $S_2^{2-}$  in pyrite, bulk disulphur, adsorbed xanthate or polysulphide, elemental sulphur or higher order polysulphide, respectively (Wagner, 1994). The broad peak at around 168 eV is representative of sulphony species (sulphate, thiosulphate) and has a higher intensity at the positive Eh value. The two components in the O 1s XPS spectra (not shown) represent oxide, hydroxide and sulphony species (Jones et al., 1992). No adsorbed water was detected on the pyrite surface (Wagner, 1994). Only one component at 932.0 eV is observed in the Cu 2p spectra (not shown). It is attributed to copper in a Cu(I) state (Wagner, 1994). No or little Cu(II) species such as copper oxide/hydroxide were observed, in agreement with the EDTA extraction results in Table 2.

The results of this spectral deconvolution shown in Table 3 indicate firstly that more xanthate is adsorbed at 130 mV than at -60 mV, in agreement with the UV-visible results in Figs. 2 and 3. Secondly, more oxidation species (sulphony and iron oxide/hydroxide species) are found on the pyrite surface at the positive Eh value, in agreement with the EDTA extraction results in Table 2. These oxidation species are likely to cover the sulphide species (decrease in the proportion of  $S_2^{2-}$ ,  $Sn^{2-}$  and elemental sulphur), the iron sulphide species (decrease from 2.0% to 1.0%) and the Cu(I) species (decrease from 1.7% to 0.7%) underneath as it was observed in our earlier XPS investigation of the oxidation of chalcopyrite (Fairthorne et al., 1997).

This study has confirmed that copper activation of pyrite is controlled by the pulp oxidation potential, Eh, and consists in the adsorption of Cu(II) species on the pyrite surface and its reduction to a Cu(I)S species. Xanthate has a high affinity for copper and its adsorption on pyrite increases with Eh values. Both the Cu(I)S and Cu(I)-xanthate species are hydrophobic and therefore responsible for the increase in pyrite flotation close to neutral Eh conditions. With Eh increasing, the pyrite oxidises and forms predominantly iron oxide/hydroxide on its surface with a small amount of sulphony species. As these species are hydrophilic, pyrite flotation decreases at Eh values larger than 50 mV. We note that pyrite recovery increases sharply with Eh, especially around a neutral Eh value, and does not appear to increase linearly with surface hydrophobicity (concentration of hydrophobic species). Mineral flotation seems to only occur above a certain threshold of surface hydrophobicity in agreement with results reported in a study on the comparison between mineral flotation and contact angle (Prestidge and Ralston, 1996).

#### 4. Conclusions

The results obtained in this study indicate that the pulp oxidation potential, Eh, is one of the key factors controlling the flotation of copper-activated pyrite as it affects the adsorption of collector but also the formation of surface oxidation products. In the conditions used in this study, a maximum recovery of copper-activated pyrite was obtained at 35 mV (SHE). The lower pyrite recoveries at Eh values lower than 7 mV are due to less SIPX adsorbed on pyrite while at Eh values higher than 50 mV the lower pyrite recoveries are attributed to more iron oxidation species present on the pyrite surface. Therefore, the overall pyrite recovery reflects the various proportions of hydrophobic and hydrophilic species present on the pyrite surface.

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